WATER AND GAS ABSORPTION OF POLYTETRAFLUOROETHYLENE AS A PIPELINE

By

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22742

Dissertation submitted in partial fulfilment of

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the

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Approved by,

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January 2020

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or undone by unspecified sources or persons.

Kuang Zhi Wen

KUANG ZHI WEN

ABSTRACT

Polytetrafluoroethylene is a material which exhibits good properties such as high melting point, low coefficient of friction, thermally stable, high electrical resistance, and good chemical inertness. Due to these properties, polytetrafluoroethylene has been widely applied in various fields such as automotive industries, biomedical and pharmaceutical applications, chemical industries, electrical applications, defense application, petrochemical industries, food processing and aerospace technology. Currently, there is limited studies on suitability of polytetrafluoroethylene as a pipeline material for hydrocarbon transportation. This project will focus on study of water and gas absorption characteristics of polytetrafluoroethylene as a pipeline and the effect to weight changes and appearance of polytetrafluoroethylene after immersed and exposed to various mediums and conditions. Water absorption of polytetrafluoroethylene is examined using common medium found causing corrosion of pipeline during hydrocarbons transportation, which are CO_2, acetic acid, and sea water. The temperature and pressure for transporting different hydrocarbon types can be vary based on their number of hydrocarbon chain. Normally, hydrocarbon transportation will be conducted with suitable temperature and pressure based on hydrocarbon properties such as wax appearance temperature, pour point temperature and hydrate formatting temperature and pressure. These properties can be obtained from on-site lab test. In this study, water absorption test and gas exposure test are carried out. Water absorption test is conducted to measure the weight changes of specimens before and after the immersion in deionized water (7.00 pH), nitric acid (0.55 pH), acetic acid (2.52 pH) and sea water (8.22 pH) after a specific period. Gas exposure test is conducted with following conditions: 1) room temperature and 1 Bar pressure, 2) room temperature and 20 Bar pressure, 3) 45 ^{o}C temperature and 20 Bar pressure using autoclave and MiCorr for a specific period. Effect of water absorption and gas exposure to the weight changes and appearance of polytetrafluoroethylene specimens are observed and recorded.

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Chapter 1
Introduction

1.1 Background

Flow assurance in pipelines and flowlines is one of the important study area for oil and gas industry. Hydrate formation, wax formations, asphaltene formation, emulsion, liquid slugging, scale, sand erosion and corrosion are the common issues found causing the leakage and failure of pipelines and flowlines during transportation of hydrocarbons. These issues can strongly impact on the oil and gas transportability thru pipelines and flowlines.

Flowlines and pipelines are different. Typically, flowlines are pipes which carrying multiphase flow from wellhead to gathering station and pipelines are pipes which carrying single-phase flow from gathering stations to processing stations or to market. Pipelines can be either carrying hydrocarbon or natural gas which is already separated by separator while in gathering station.

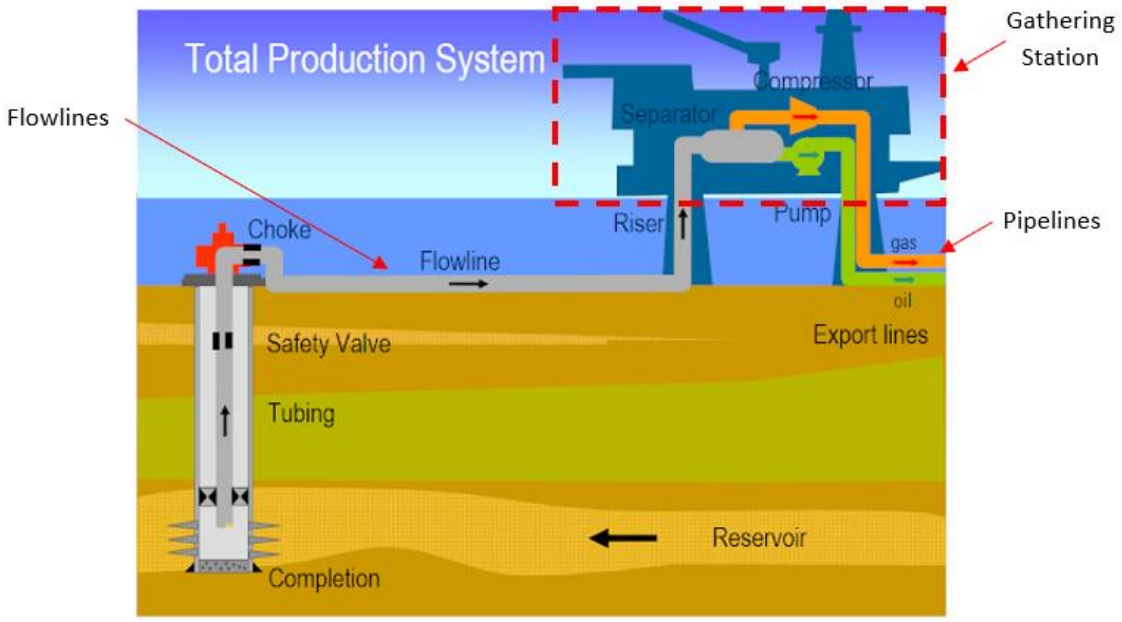

Figure 1.1 Illustration of flowlines and pipelines in oil and gas production system. [1]

1.2 Problem Statement

Leakage and failure of a pipeline need to be prevented to ensure the flow assurance of hydrocarbons. Based on a report from Canadian Association of Petroleum Producers (CAPP) on failure statistics of pipeline incident happening in Canada since 1985 to 2008, internal corrosion is the most dominant reason for leakage and failure for natural gas pipeline and sour gas pipeline [2]. As most of the pipeline used in oil and gas industries are fabricated from carbon steel and low alloy steel grade, corrosion of metallic pipelines is one common issue facing by these oil and gas industries during their operational activities.

To tackle down the corrosion issue, using pipeline which is fabricated by non-metallic material with good corrosion resistance could be one of the potential solutions. Polytetrafluoroethylene is a non-metallic material which exhibits good corrosion resistance. However, as the material need to be constantly immersed and exposed to various mediums and conditions if it is applied in hydrocarbons transportation activities as a pipeline. The effect of water and gas absorption of polytetrafluoroethylene when immersed and exposed to various mediums and conditions found during hydrocarbons transportation activities need to be study.

1.3 Objective

In this project, non-metallic material - polytetrafluoroethylene is chosen to be study. The objective of this project is:

1) To evaluate the water absorption of polytetrafluoroethylene when immersed in deionized water, sea water, acetic acid and nitric acid for a specific period.
2) To evaluate gas absorption of polytetrafluoroethylene after some period of exposure to different temperature and different pressure conditions.
3) To observe and record the changes in weight and physical characteristics of polytetrafluoroethylene after immersed and exposed to various conditions.

1.4 Scope of Study

The aim of this project is to study the water and gas absorption of polytetrafluoroethylene and the effect to its weight and appearance after exposed to mediums and conditions which is commonly found causing the corrosion and failure to metallic pipeline during oil and gas operational activities, as shown in Table 1.1.

TABLE 1.1 Test Matrix.

Testing Method	Medium	pH	Condition	Equipment	Test Length (Hour)	Sample Label
Water Absorption Test	Deionized Water	7.00	Room Temperature & Room Pressure	-	336	A21 – A25
	Sea Water	8.22	Room Temperature & Room Pressure	-	336	B21 – B25
	10% Acetic Acid	2.52	Room Temperature & Room Pressure	-	336	C21 – C25
	10% Nitric Acid	0.55	Room Temperature & Room Pressure	-	336	H21 – H25
Gas Exposure Test	CO_2 gas + Water + Hydrocarbon (50% volumetric fraction of Deionized water)	-	25^{o}C, 1 bar	MiCorr	168	D21 – D25
			25^{o}C, 20 bars	MiCorr	168	F21 – F25
			45^{o}C, 20 bars	Autoclave	120	G21 – G25
Control Unit	-	-	Room Temperature & Room Pressure	-	336	E21 – E25
Total Sample Required						40

Chapter 2
Literature Review

2.1 Corrosion of Metallic Pipeline in Oil and Gas Industry

Pipelines has been incorporated in oil and gas industry to serve the purpose of transporting hydrocarbon and products since 19th century [3]. As oil and gas industries getting more matured, many studies have been done on the corrosion of metallic pipeline and many standards has been introduced for corrosion control.

Metallic pipelines use in oil and gas industries are mainly manufactured from raw material which is carbon steel and alloy steel. However, corrosion will happen to metallic pipeline if the surface of the metallic pipeline is exposed to the corrosive environment. Moreover, as the inner wall and outer wall of the pipeline is facing at a different environmental condition, thus, corrosion occurs in internal surfaces and external surfaces will have different scenario. Also, the technique used for external corrosion control cannot be effective to internal corrosion control.

Several parameters can influence the corrosion of a pipelines, such as H_2S and CO_2 concentration, chlorine ions concentration, existing of organic acids, flow regime, water cut, temperature and pressure. Due to complexity of the interference between all the parameters, only the mechanisms of the corrosion will be discussed in this literature review.

Pipeline corrosion can cause metal loss and result in thinner wall. Type of corrosion which is caused by different factors can be categorized into different mechanism of corrosion. Each mechanism of corrosion will result the corroded surface with different features and configuration.

Failure of a pipeline is always caused by two important factors, which are general and localized corrosion and environmentally assisted cracking. They can be further breakdown into more type details of corrosion and cracking, as shown in Figure 2.1 and 2.2.

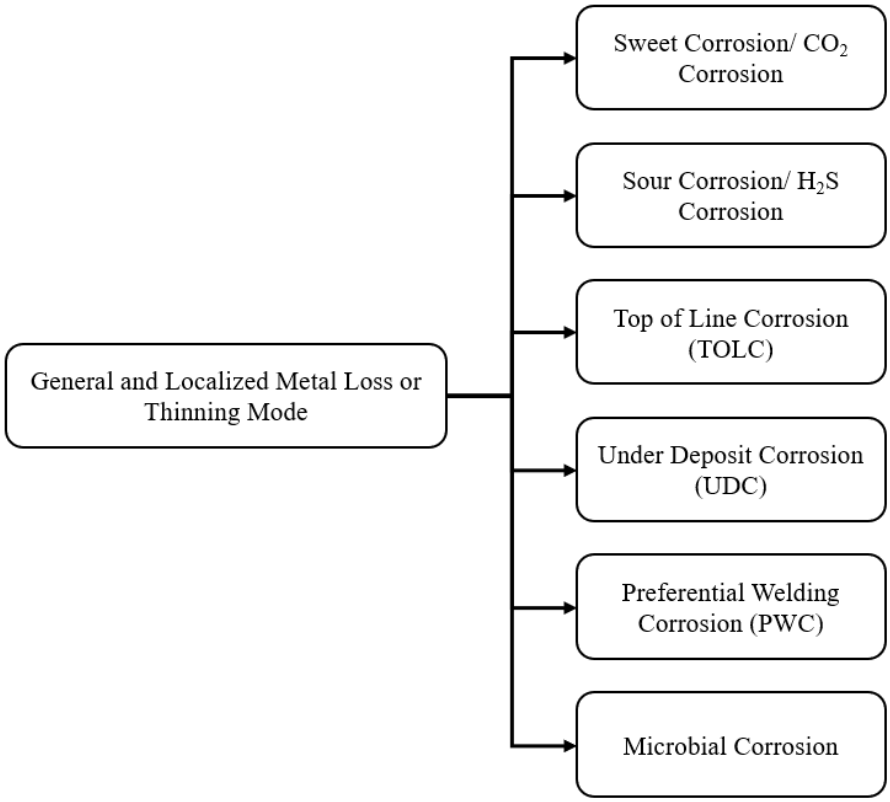

Figure 2.1 Types of general corrosion and localized corrosion.

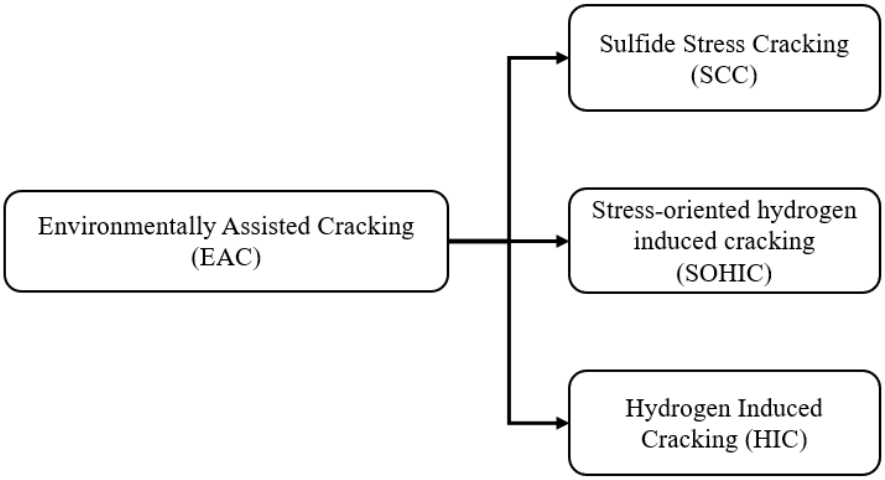

Figure 2.2 Types of environmentally assisted cracking.

Illustration of different forms of internal corrosion occurring in hydrocarbon pipelines are as shown in Figure 2.3.

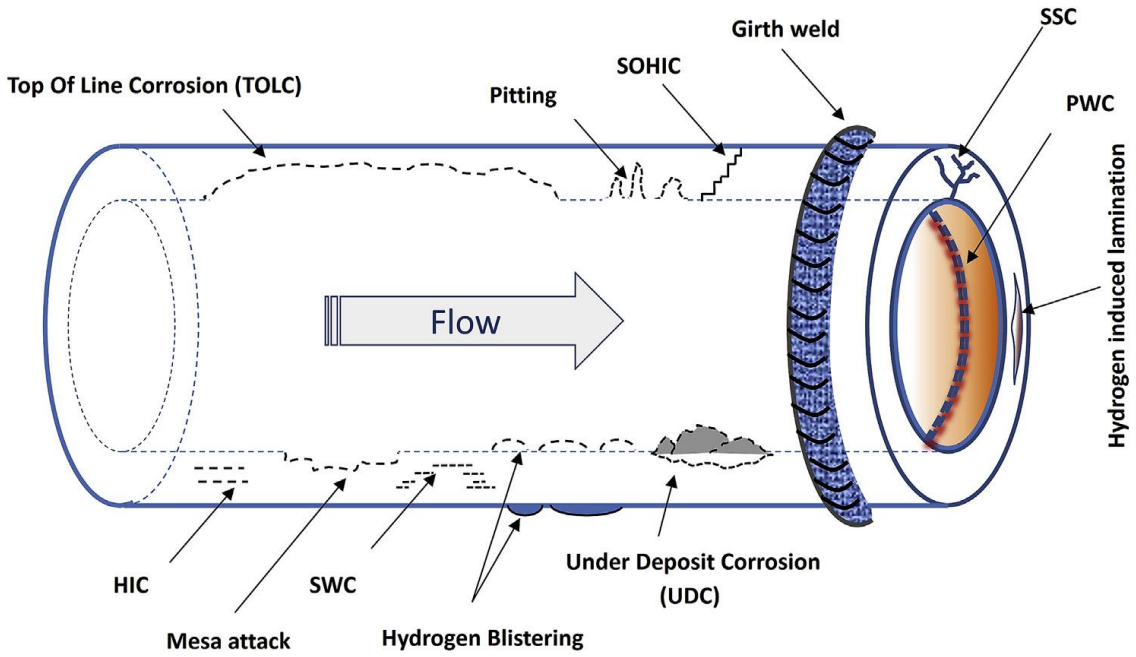

Figure 2.3 Different forms of internal corrosion in hydrocarbon pipelines. [2]

2.1.1 CO_2 Corrosion Mechanism/ Sweet Corrosion Mechanism

CO_2 is present in oil and gas reservoirs and is soluble in water and brine. As CO_2 dissolved into water and brine, it became acidic and can cause corrosion to metal. CO_2 corrosion occurs more in the form of general corrosion and is the most common type in pipeline. Despite, CO_2 can also result in localized mode failure with three variants which is pitting, mesa attack and flow induced.

In general, CO_2 dissolves in water and brine to give carbonic acid refer to Equation 1.

$$CO_2 + H_2O \rightleftarrows CO_2 - H_2O \simeq H_2CO_3 \rightleftarrows H^+ + HCO_3^- \qquad (1)$$

Throughout the studies on CO_2 corrosion occuring in pipeline by researchers, several chemical reaction which may possibly cause the corrosion of pipeline has been proposed by researchers, as listed in Table 2.1.

TABLE 2.1 Chemical Reactions for CO_2 Corrosion. [4]

Chemical Reaction		Reaction proposed by	Year
(HCO_3^-): $H^+ + e^- \rightarrow H$, $2H \rightarrow H^2$	**(2)**	Schwen Kermani and Smith [13, 20]	1974 1997
$H_2CO_3 + e^- \rightarrow H + HCO_3^-$	**(3)**	De Waard and Milliams [4]	1975
$HCO_3^- + H+ \rightleftarrows H_2CO_3$, $2H \rightarrow H_2$	**(4)**		
$HCO_3^- + e^- \rightarrow H + CO_3^{2-}$	**(5)**	Ogundele and White [16]	1987
$HCO_3^- + H + e^- \rightarrow H_2 + CO_3^{2-}$	**(6)**		

Regardless of whichever chemical reaction is correct, all the chemical reactions in Table 2.1 showing that CO_2 is one of the factor causing the corrosion to be happened in metallic pipeline [2, 4, 5, 6].

Different corrosion types induce by CO_2 in multiphase flow during transportation of hydrocarbons and products can be categorized as shown in Figure 2.4.

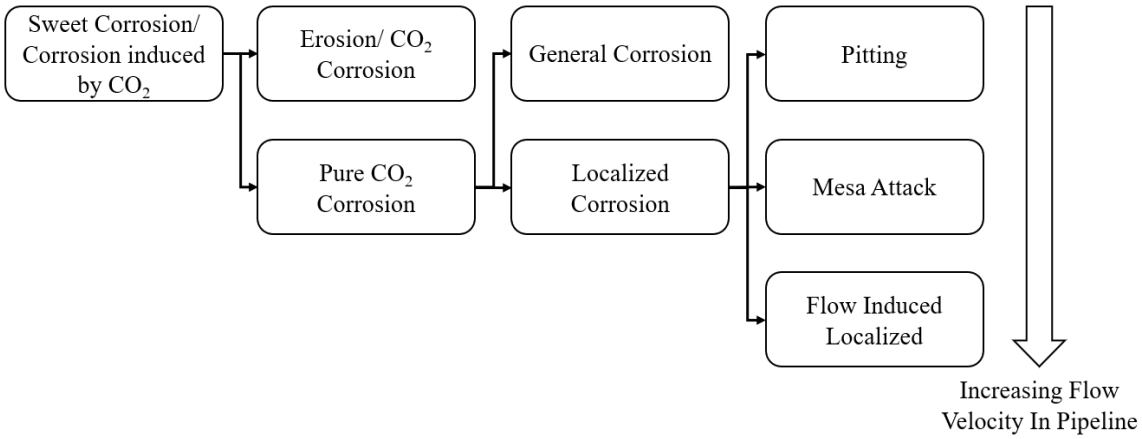

Figure 2.4 CO_2 corrosion in different formation.

2.1.1.1 General Corrosion

According to the electrochemical reaction proposed by Nesic [2], corrosion mechanism of general corrosion is due to reaction between metal loss from pipeline (Fe) and acidic solution (CO_2 dissolved into water). The product of this reaction is carbonate scales and hydrogen gas, as shown in Equation 7.

$$Fe + CO_2 + H_2O \rightarrow FeCO_3 + H_2 \quad (7)$$

2.1.1.2 Localized Corrosion

On top of general corrosion, relationship between corrosion form of metallic pipeline and fluid flow velocity is also studied by researchers. This kind of corrosion is named as localized corrosion. Different form of corrosion is observed when there is changes in fluid flow velocity, and they are differentiated into categories of pitting corrosion, mesa attack and flow induced localized corrosion.

2.1.1.2.1 Pitting Corrosion

Pitting corrosion occurs in pipeline where the flow velocity of fluid is low and around the dew point temperatures. Rate of pitting corrosion is likely to be increased when temperature and partial pressure of CO_2 is increased.

2.1.1.2.2 Mesa Attack

Mesa attack has been observed when flow velocity of fluid in a pipeline is in range of low to medium velocity. Flow regime of fluid in this range of velocity can caused damage to the carbonate scale on the inner surface of the pipeline, thus causing the carbonate scale protective film to be breakdown. After some studies, researchers also concluded that the mesa attack corrosion rate at area which is not well covered by $FeCO_3$ carbonate scale film is much higher than at area which is well covered by $FeCO_3$ carbonate scale film. $FeCO_3$ carbonate scale is a product formed during general corrosion [2, 4].

2.1.1.2.3 Flow Induced Localized Corrosion

When flow velocity of fluid in a pipeline is high and reach above 10 m/s, form of corrosion will change from mesa attack to flow induced localized corrosion. Due to higher velocity, more carbonate scale film from the pipe wall will be breakdown. Higher velocity can trigger the turbulence effect of the fluid flow, which causing more stresses apply on the carbonate scale film. After the protective film has been breakdown, the new formation of carbonate scale will obstruct by the high velocity fluid, causing the carbonate scale film cannot reform on the pipe surface to protect it from corrosion. Therefore, the corrosion to the metallic pipe wall will occurs in high rates.

Besides, uneven surface of the pipe inner wall after formation of carbonate scale film can also cause microturbulences when velocity of flow is high. This will lead to thinning of protective film and growth of porosity. Eventually, this can cause destruction of the film, as shown in Figure 2.5.

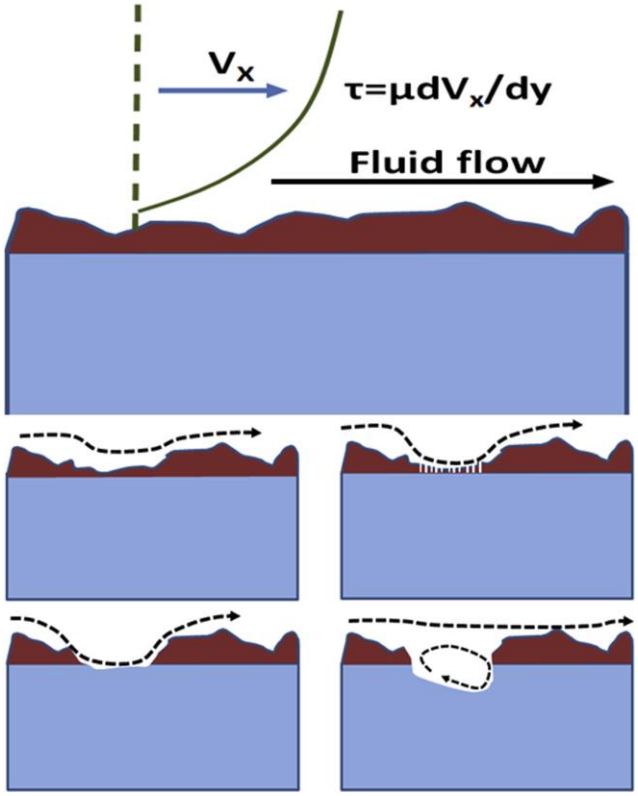

Figure 2.5 Development of flow induced localized corrosion. [2]

2.1.2 H_2S Corrosion Mechanism/ Sour Corrosion Mechanism

For sour corrosion, H_2S is the main factor causing the corrosion of a pipeline. H_2S is an acidic gas which can be dissolved in multiphase flow of hydrocarbons. Generally, as H_2S is dissolving into the fluid, it will be dissociated and produce H^+ cations. Next, the produced H^+ cations will capture electrons and cause electrochemical reaction between the H^+ cations and metal loss from pipeline, thus result in thinning of metallic pipeline, which is corrosion.

The chemical reaction for H_2S corrosion in multiphase flow is as shown in Equation 8.

$$Fe + H_2S \rightarrow FeS + H_2 \qquad (8)$$

Due to the complicated chemistry of H_2S - H_2O system, exact mechanism of H_2S corrosion is currently still difficult to be determined by researchers.

2.1.3 Microbiologically Influenced Corrosion Mechanism

In operational activities, microbiologically influenced corrosion is having potential to cause internal corrosion and failure of a pipeline. Several factors such as sulfate concentration, nutrients concentration, type of bacteria, pH value of fluid, fluid flow rate, salinity of solution, and surrounding temperature can affect the corrosion rate of microbiological influenced corrosion. Normally, these bacteria are classified as either aerobic or anaerobic.

During the microbial activities, some chemicals are produced by the bacteria. These chemicals produced by the bacteria may react with other elements in the surrounding and form an acid. Thus, the acidic environment can cause an acceleration to the corrosion rate of metallic pipeline. Most of the case, microbiological influenced corrosion is found in localized corrosion configuration.

Figure 2.6 shows the major microbial species which are influencing on MIC.

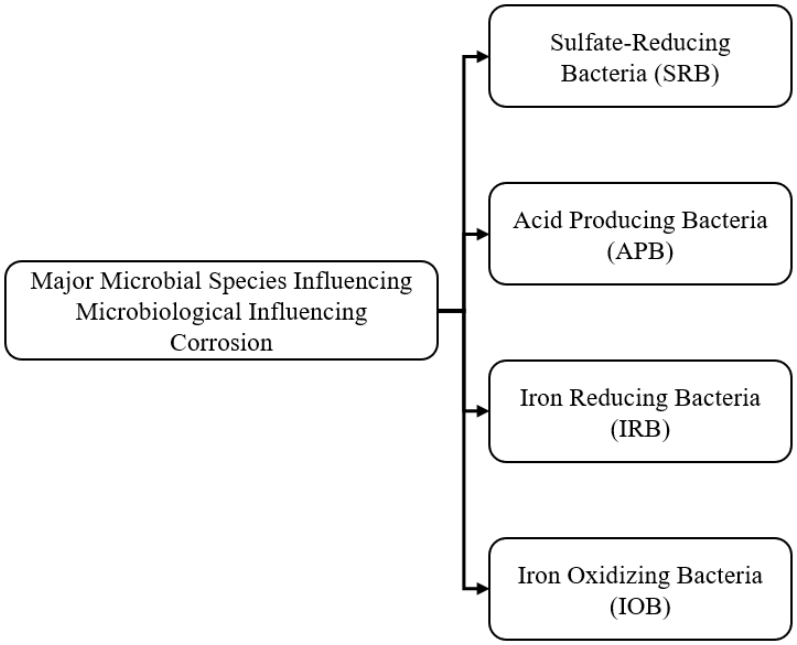

Figure 2.6 Major microbial species influencing MIC.

2.1.3.1 Sulfate-Reducing Bacteria (SRB)

Sulfate-reducing bacteria (SRB) reduces sulfate to hydrogen sulfide to obtain their energy as shown in Equation 9. Increasing rate of hydrogen reduction lead to faster production rate of corrosion product, which is FeS. The chemical reaction for formation of FeS is as shown in Equation 10.

$$SO_4^{2-} + CH_3COOH + 2H^+ \rightarrow HS^- + 2HCO_3^- + 3H^+ \quad (9)$$

$$HS^- + Fe^{2+} \rightarrow FeS + H^+ \quad (10)$$

2.1.3.2 Acid Producing Bacteria (APB)

Working principle of acid producing bacteria (APB) is to convert organic materials into CO_2. The produced CO_2 is then dissolved into the fluid and causing the fluid to be acidic, thus causing general corrosion and localized corrosion of the pipeline.

2.1.3.3 Iron Reducing Bacteria (IRB)

Iron reducing bacteria (IRB) reduce Fe^{3+} to soluble iron ions which is Fe^{2+}, this will cause the insoluble film on the pipe surface to be reduced to soluble film, as shown in Equation 11. Thus, the electrochemical reaction can happen between reagents and the film [7]. Electrochemical reaction between reagents and film can cause thinning of film and eventually cause the metal surface to be exposed to the corrosive medium. Moreover, IRB can also create anaerobic zones which is suitable for growth of sulfate reducing bacteria within biofilm and induce different combination of corrosion.

$$4H_2O + 2Fe_2O_3 \rightarrow 4Fe(OH)_2 + O_2 \quad (11)$$

2.1.3.4 Iron Oxidizing Bacteria (IOB)

Different from iron reducing bacteria (IRB), soluble Fe^{2+} will be oxidizes into insoluble Fe^{3+} by iron oxidizing bacteria (IOB) and form an insoluble layer on the surfaces of internal pipe wall, as shown in Equation 12 [6]. The newly formed insoluble layer will have rough and uneven surfaces. Due to the rough and uneven surface of the insoluble layer, the layer can be damaged by high velocity turbulence flow and microturbulence flow when fluid flow velocity is equal or above 10 m/s. Once the insoluble layer is break down, metal surface will again be exposed to the corrosive medium. Thus, localized corrosion will occur.

$$4Fe(OH)_2 + O_2 \rightarrow 4H_2O + 2Fe_2O_3 \quad (12)$$

2.1.4 Top of the Line Corrosion (TOLC)

Main reason causing top of the line corrosion is due to water condensation at the top of pipeline internal surface. Rapid drop in temperature to below dew point will cause water to easily condense at the inner of pipeline surface, which has potential causing this

kind of corrosion. Besides, there are also several factors which can affect the condensation rate of water, as shown in Figure 2.7.

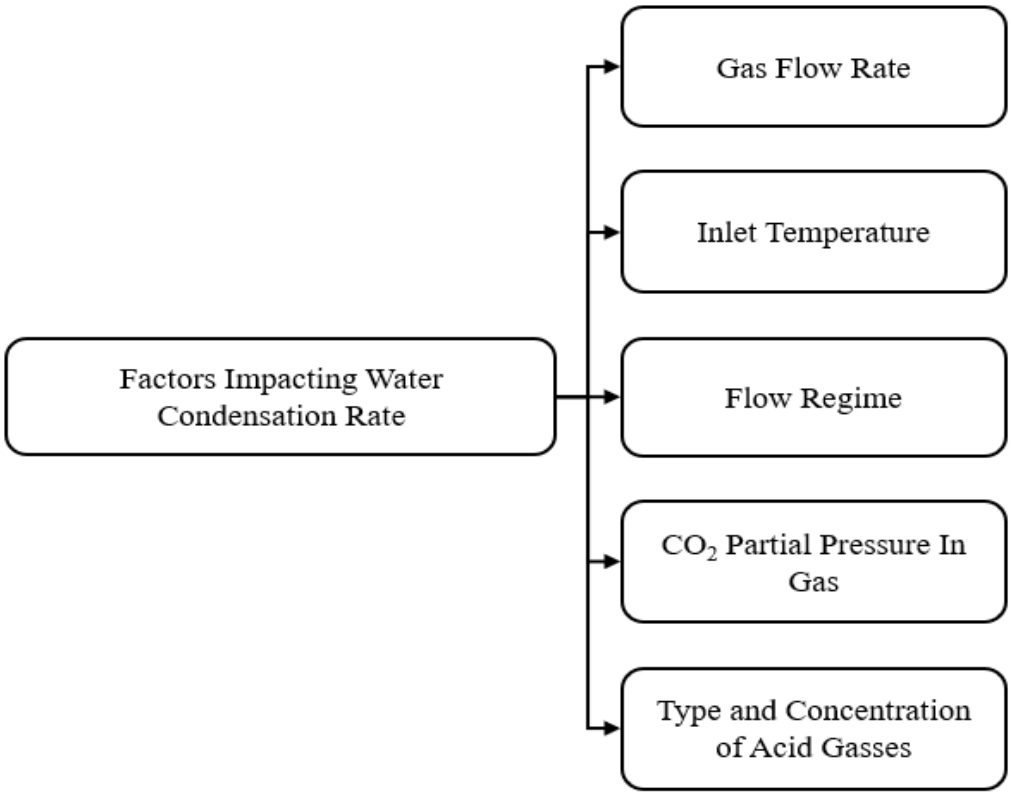

Figure 2.7 Factors impacting water condensation rate.

Commonly, top of the line corrosion is most likely happening in pipeline which use for transporting sweet hydrocarbon containing 500 to 3000 ppm of organic acid such as acetic acid, formic acid, propionic acid and butanoic acid [8]. The corrosive agents to initiate the corrosion is believed to be CO_2 and organic acid.

Figure 2.8 is showing the formation of top of the line corrosion. Condensed water droplets and wet film can form when pipeline has poor insulation or section of pipeline is exposed to surrounding. Condensed water droplets and wet film are form due to the temperature differences between buried section of pipeline and unburied section of pipeline.

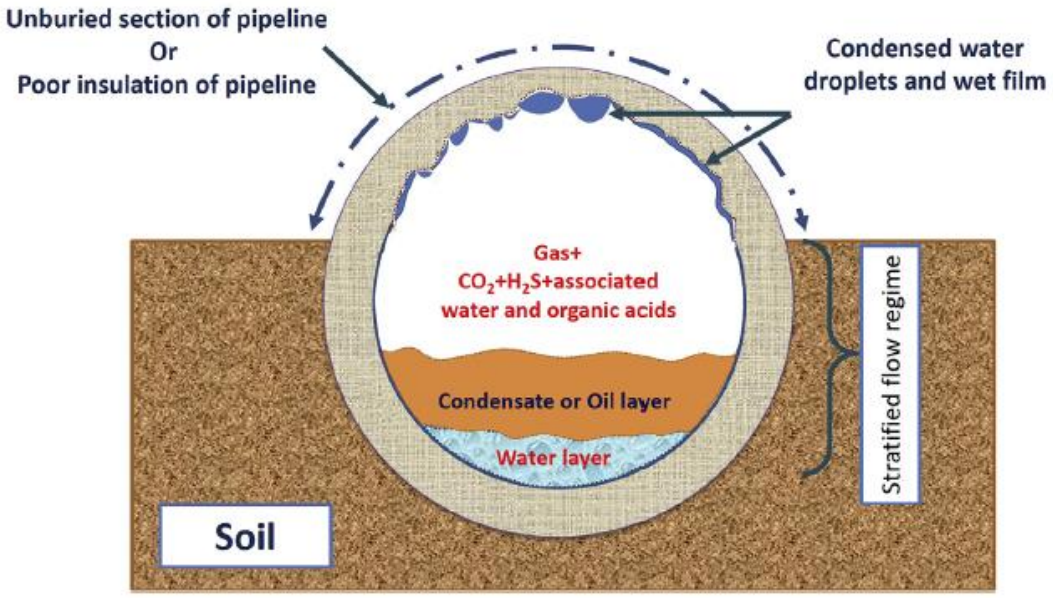

Figure 2.8 Condensation and TOLC in unburied section of pipeline. [2]

2.1.5 Under Deposit Corrosion

During transportation of hydrocarbons, impurities containing will be deposited along the way of pipelines. These impurities consist of alumina, hydrated alumina, calcite, clays, iron scales, silica, corrosion products and organic compounds. As impurities accumulated, bed of solids will be formed at the bottom of the pipelines and prevent direct contact of corrosion inhibitor with the pipeline surface. Eventually, localized attack can occur.

Under deposit corrosion is likely to occurs in horizontal pipelines instead of vertical pipeline especially when the fluid flow velocity is low. In this case, the formation of solid beds of impurities is unable to be obstruct by either the low velocity fluid flow or gravitational force acting in vertical direction.

2.1.6 Preferential Weld Corrosion

Weld area and heat-affected zone is observed more active to be corroded compare to non-heat affected zone when both locations are exposed to corrosive medium with environment conditions of high temperature and high flow rate.

However, the exact mechanism of preferential weld corrosion is still unclear. One possible idea which is widely accepted by researchers is that, weld or heat affected zone (HAZ) area would be anodic to its parent material, thus the localized corrosion can be happen due to galvanic effect [2].

2.1.7 Environmental Assisted Cracking by wet H_2S

In wet sour gas pipeline, H_2S can have corrosion reaction with metal ions to produce atomic hydrogen as a byproduct, as shown in Equation 13.

$$Fe + 2H^{+} \rightarrow Fe^{2+} + 2H^{0} \qquad (13)$$

Atomic hydrogen is very small in size and can easily diffuse into the carbon steel structure and trapped inside the structure and induce cracking as shown in Figure 2.9. When atomic hydrogen is recombining inside the carbon steel structure, due to the recombined molecular hydrogen will have larger molecular size, therefore causing the molecule cannot escape from the carbon steel structure. The accumulated molecular hydrogen trapped inside the carbon steel structure can build up pressure in the carbon steel structure and causing the structure to subject to bulging and blistering as shown in Figure 2.10 [2, 9].

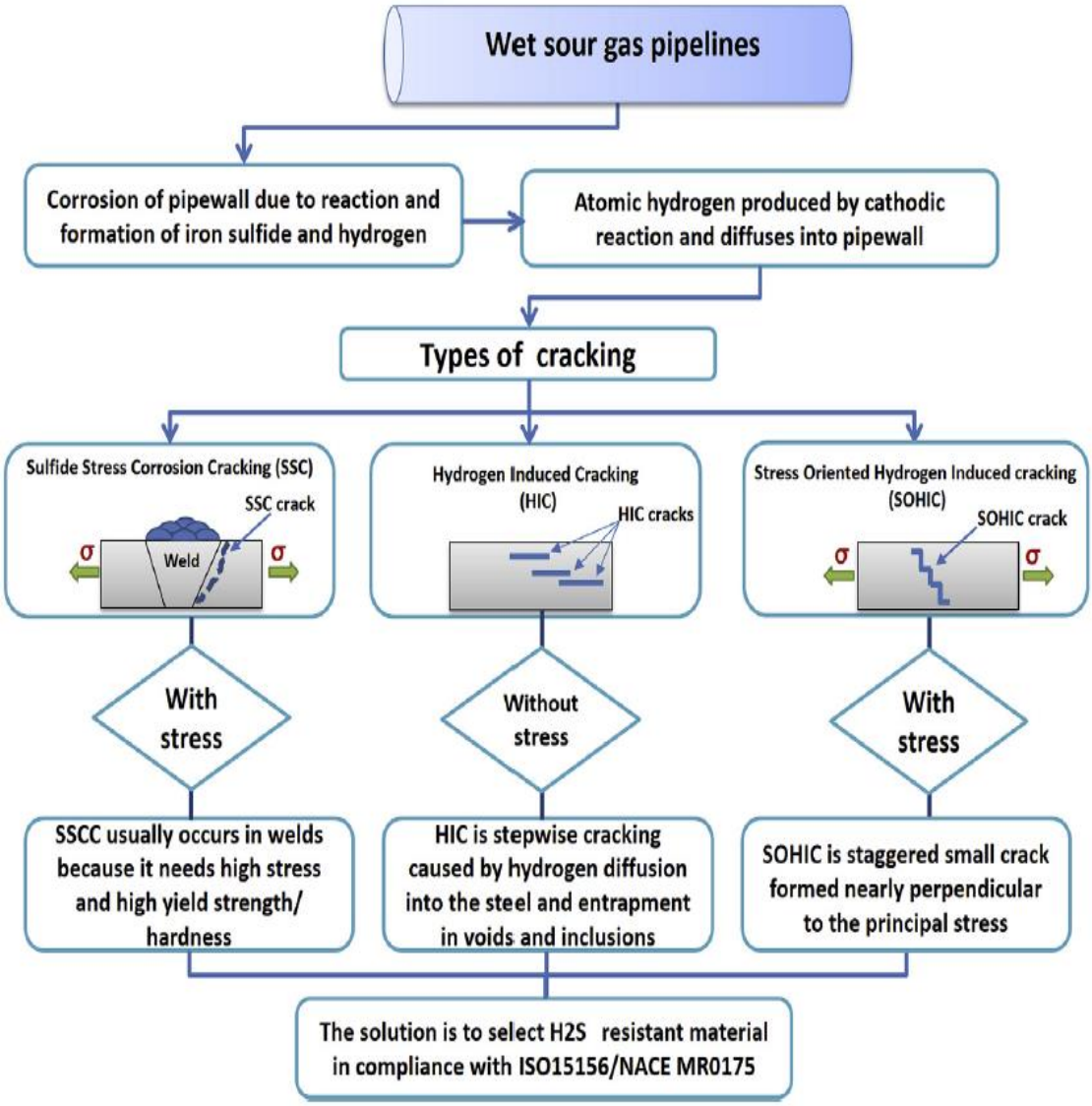

Figure 2.9 Major cracking damages by H_2S in oil and gas pipelines. [2]

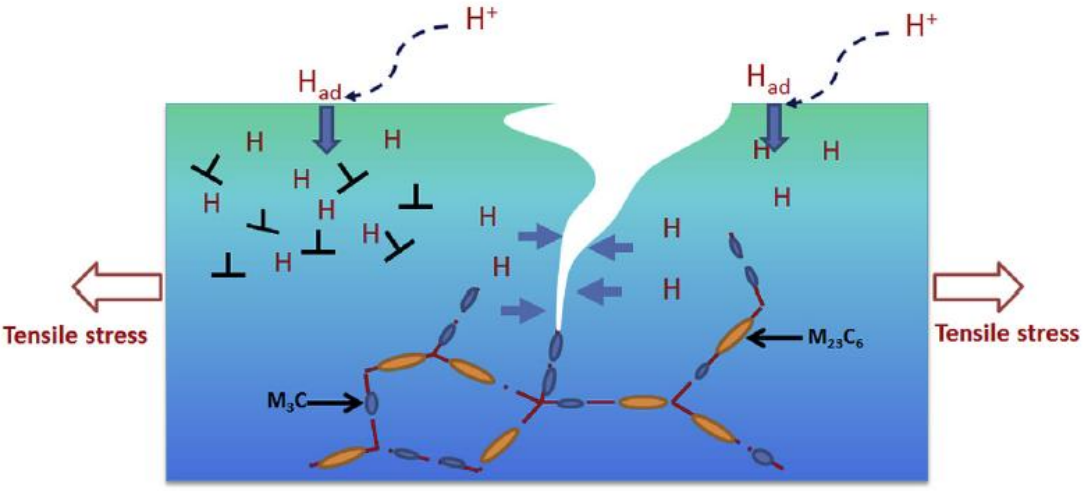

Figure 2.10 Schematic diagram of development of environmental assisted cracking. [2]

2.2 Polytetrafluoroethylene: Structure and Its Properties

2.2.1 Structure of Polytetrafluoroethylene

Polytetrafluoroethylene (PTFE) is a fluoropolymer, was first accidentally discovered by Dr. Roy J. Plunkett at the DuPont industry in 1938. Studies have been done by researchers on its structure. Polytetrafluoroethylene is manufacturer when the monomer Tetrafluoroethylene (TFE) undergoes free radical vinyl polymerization [10, 11, 12].

The name of TFE is combination of three terms, which is “Tetra”, “Fluoro” and “Ethylene”. “Tetra” means there are four atoms attracted to the carbons. “Fluoro” means the attracted atoms are fluorine. “Ethylene” means the carbons are attached to each other as classic ethylene structure. TFE is having molecular formula of C_2F_4. When TFE is polymerizes into PTFE, the double bond between carbons will reformate into single bond. PTFE is consisting of long chain of carbon atoms.

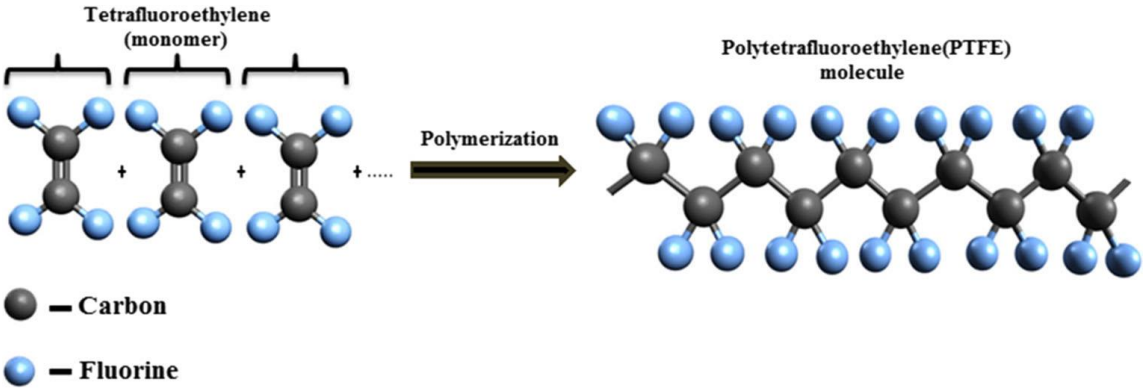

Fig. 2.11. Change in molecular structure when TFE polymerizes into PTFE. [11]

PTFE possesses C-F bond in the formulation and having molecular formula $[(CF_2\text{-}CF_2)_n]$. Due to the strongly attached bond between fluorine and carbon atoms, PTFE exhibits properties such as high molecular weight and chemical inertness.

2.2.2 Properties of Polytetrafluoroethylene

Material datasheets of PTFE from different raw material manufacturer are being compared and review. The test value listed in below table is tested by raw material manufacturer according to either International Organization of Standardization (ISO) or American Society for Testing and Materials (ASTM) which the value obtained is reliable.

TABLE 2.2 Comparison of Test Value Between Raw Material Manufacturer.

Properties	Unit	Raw Material Manufacturer and Test Standard							
		Test Standard	TEKNIK srl	Test Standard	DuPont de Nemours Inc.	Test Standard	Foresight Plastics and Engineering	Test Standard	Standard Fluoromers Pvt. Ltd
Density	g/cm^2	ISO 13000-2	2.130 – 2.180	ASTM D792	2.16	ASTM D792	2.16 – 2.20	ASTM D792	2.1 – 2.2
Tensile Strength	MPa	ISO 13000-2	≥ 20	ASTM D638	26.89	ASTM D638	≥ 20	ASTM D638	20.59 – 36.77
Elongation	%	ISO 13000-2	≥ 200	ASTM D638	300	ASTM D638	≥ 300	ASTM D638	250 - 400
Hardness	Shore D	ISO 13000-2	≥ 54	ASTM D785	50	ASTM D785	50 - 55	ASTM D2240	58 - 62
Thermal Conductivity	W/m K	ASTM C177	0.24	ASTM C177	0.245	ASTM D2214	0.2	-	-
Melting Temperature	°C	-	-	ASTM D3418	335	-	-	-	-
Water Absorption, 24 hrs	%	-	-	ASTM D570	< 0.01	ASTM D570	< 0.01	ASTM D570	0
Permeability	%	-	-	-	-	-	-	ASTM D543	0.01
Dissolution	%	-	-	-	-	-	-	ASTM D543	0.01

Chapter 3
Methodology/ Project Work

By understanding the basic corrosion contributor to metallic piping system during production of oil and gas industry, deionized water, sea water, CO_2, acetic acid and nitric acid are used to study the corrosion of polytetrafluoroethylene. The process flow of the project is arranged as shown in Figure 3.1.

3.1 Process Flow

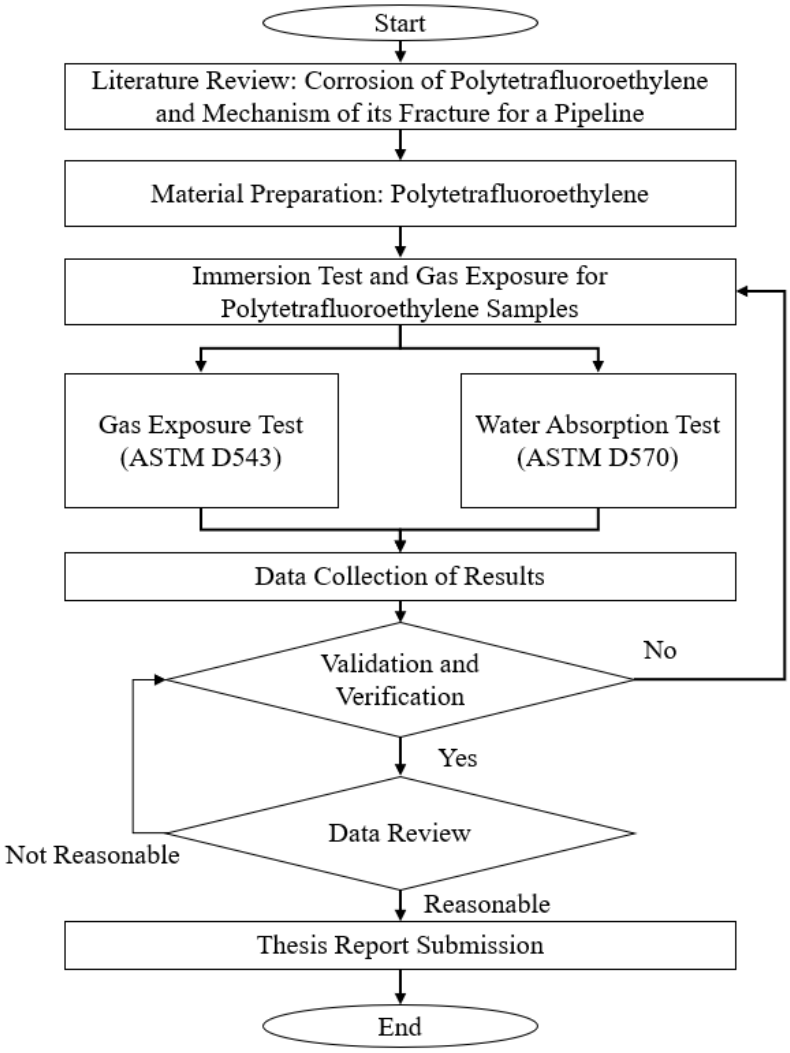

Figure 3.1 Process flow of the study.

3.2 Test Specimen Preparation

Before starting the experiment, raw material – PTFE sheet (as shown in Figure 3.2) is prepared.

Figure 3.2 PTFE sheet with dimension 1500 mm x 1500 mm x 3 mm.

Procedure:

1) PTFE sheet with dimension 1500 mm x 1500 mm x 3 mm is cut into bone-like shape specimen as shown in Figure 3.3, following Type $V^{C,D}$ specification listed in Table 3.1.

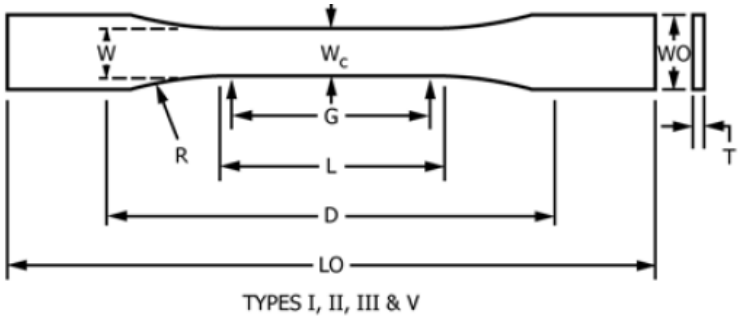

Figure 3.3 Bone-like shape specimen. (Retrieved from ASTM D638)

TABLE 3.1 Specimen dimension for thickness, T, mm (in.)[A]. (Retrieved from ASTM D638)

Dimensions (see drawings)	4 (0.16) or under		Tolerances
	Type IV[B]	Type V[C,D]	
W—Width of narrow section[E,F]	6 (0.25)	3.18 (0.125)	±0.5 (±0.02)[B,C]
L—Length of narrow section	33 (1.30)	9.53 (0.375)	±0.5 (±0.02)[C]
WO—Width overall, min[G]	19 (0.75)	...	+ 6.4 (+ 0.25)
WO—Width overall, min[G]	...	9.53 (0.375)	+ 3.18 (+ 0.125)
LO—Length overall, min[H]	115 (4.5)	63.5 (2.5)	no max (no max)
G—Gage length[I]	...	7.62 (0.300)	±0.25 (±0.010)[C]
G—Gage length[I]	25 (1.00)	...	±0.13 (±0.005)
D—Distance between grips	65 (2.5)[J]	25.4 (1.0)	±5 (±0.2)
R—Radius of fillet	14 (0.56)	12.7 (0.5)	±1 (±0.04)[C]
RO—Outer radius (Type IV)	25 (1.00)	...	±1 (±0.04)

2) Each specimens are weighted and recorded before the experiment starts.

3.3 Water Absorption Test (ASTM D570)

Polytetrafluoroethylene test specimen will be immersed into four different reagents which is sea water, deionized water, acetic acid and nitric acid for a given specific time, the result will be observed and recorded.

Procedure:

1) 1000 mL of sea water is collected from sea nearby Lumut (4°11'16.4"N 100°36'24.7"E), Perak, Malaysia. 1000 mL of deionized water is prepared by UTP laboratory. 1000 mL of 10% acetic acid is prepared by adding 96 mL (101 g) of glacial acetic acid (sp gr 1.05) to 910 mL of deionized water. 1000 mL of 10% nitric acid is prepared by adding 108 mL (153 g) of HNO_3 (sp gr 1.42) to 901 mL of deionized water.
2) Environment to carry out the immersion test is maintained at standard laboratory atmosphere of 23 ± 2 °C and 50 ± 10 % relative humidity in accordance with Practice D618.
3) Container which is resistant to the corrosive effects of the medium is used. Test specimen is immersed into medium and the container is tightly sealed to minimize loss.

4) Five test specimens are immersed into each reagent with period of 1 day, 2-days, 3-days, 7-days and 14-days.
5) Reagents is stirred every 24 hours by moderate manual rotation.
6) After the immersion of test specimen reach the specific time, test specimen is removed from reagent and immediately weighted. Next, the test specimen is wash with running water to clean from reagents. Test specimen is wipe with dry cloth or tissue and immediately weighted again.
7) Surface of the test specimen is observed.

Figure 3.4 to 3.8 shows the polytetrafluoroethylene specimens immersed in different medium such as deionized water, sea water, 10% acetic acid, 10% nitric acid. One set of control unit is prepared for comparison. Each specimens are labelled according to Table 3.4.

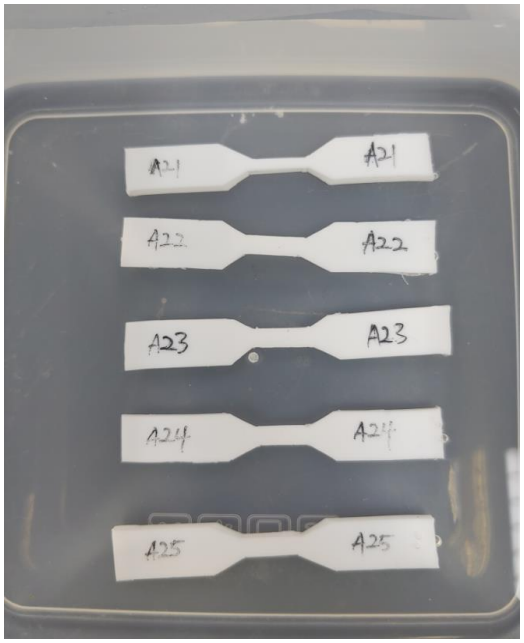

Figure 3.4 Specimens immersed in deionized water (A21 to A25).

Figure 3.5 Specimens immersed in sea water (B21 to B25).

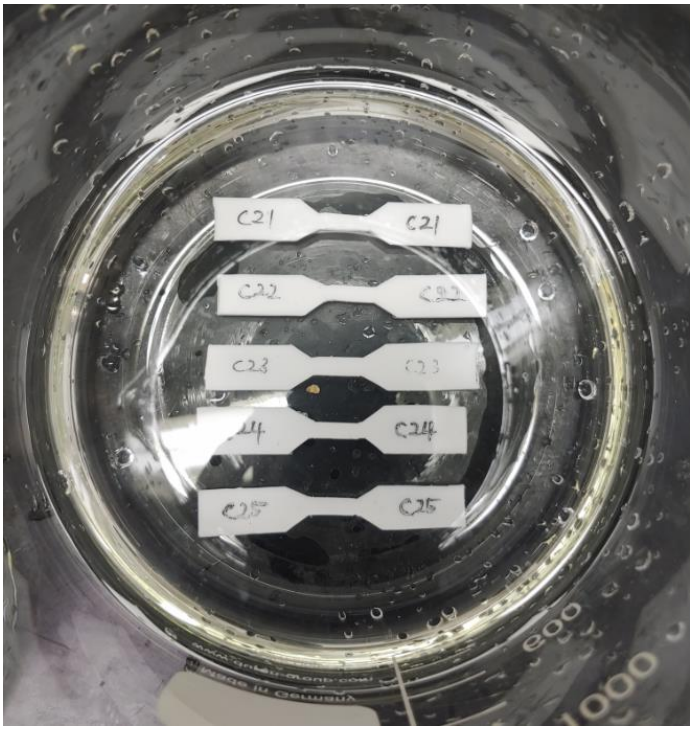

Figure 3.6 Specimens immersed in 10% acetic acid (C21 to C25).

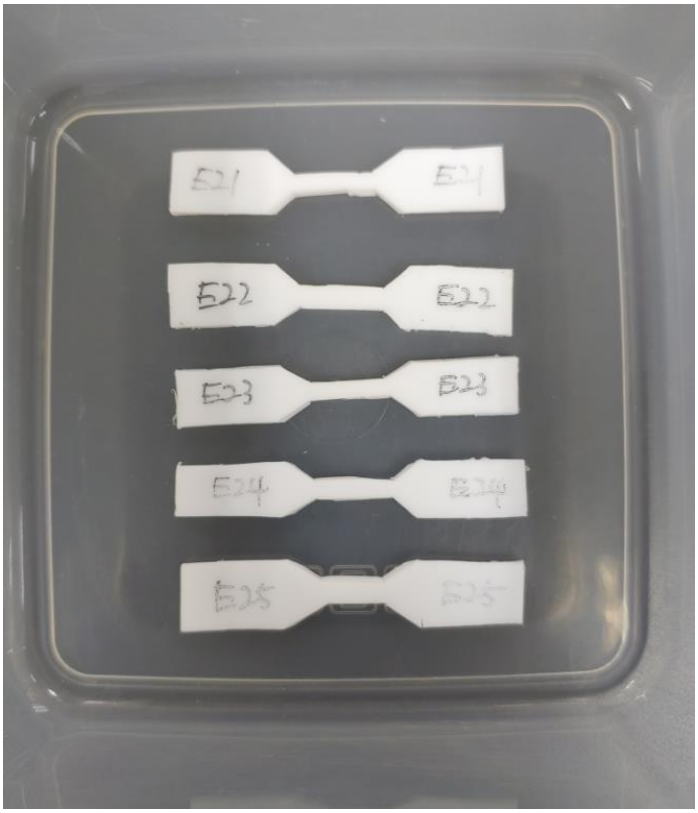

Figure 3.7 Control unit (E21 – E25).

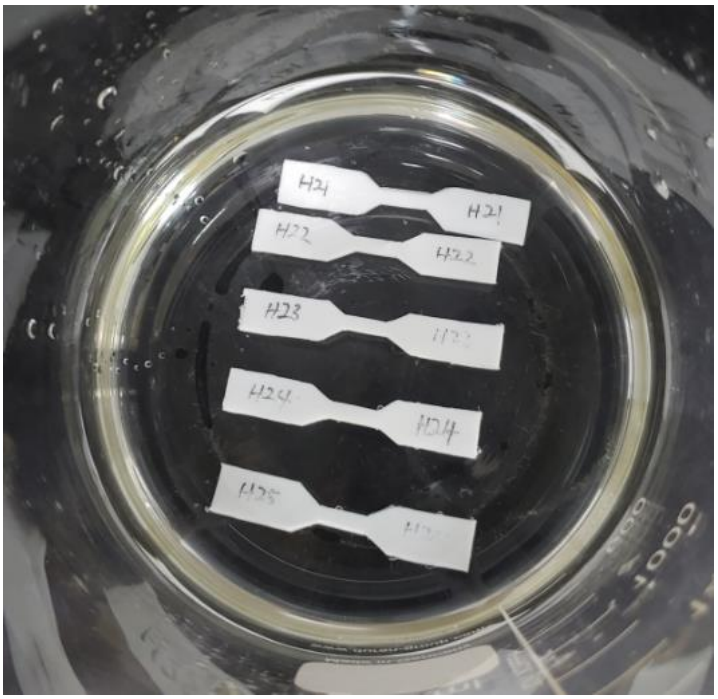

Figure 3.8 Specimens immersed in 10% nitric acid (H21 – H25).

3.4 Gas Exposure Test (ASTM D543)

Polytetrafluoroethylene test specimen will be chemically exposed to the medium of mixture with combination of CO_2 gas, deionized water and hydrocarbon with different temperature and pressure for a specific time, the result will be observed and recorded.

Procedure:

1) Test specimens are exposed to three different kind of conditions as listed in Table 3.2. Five piece of specimens are used for testing in each condition.

TABLE 3.2 Test Condition for Gas Exposure Test.

Medium	Condition	Temperature (^oC)	Pressure (bar)	Equipment	Exposure Time (hour)
CO_2 + Deionized Water + Hydrocarbon (50% Water Cut)	D	25	1	MiCorr	168
	F	25	20	MiCorr	168
	G	45	20	Autoclave	120

2) After the specified exposure period has reached, the test specimens are wash with running water to clean from medium. Test specimen is wipe with dry cloth or tissue and immediately weighted again.

3) Surface and appearance changes to the test specimen is observed and recorded.

For gas exposure test, the bone shaped polytetrafluoroethylene specimens are immersed into mix solution of 250 mL hydrocarbon and 250 mL deionized water, as shown in Figure 3.9. The solution is then poured into Mobile In-Situ Corrosion Monitoring Equipment or Autoclave as shown in Figure 3.10 and Figure 3.11.

Next, the equipment is sealed and pressurized with CO_2 gas to achieve the desired test pressure. Autoclave equipment is heated by a gray heating jacket surrounding the equipment (as shown in Figure 3.11) to achieve the desired test temperature.

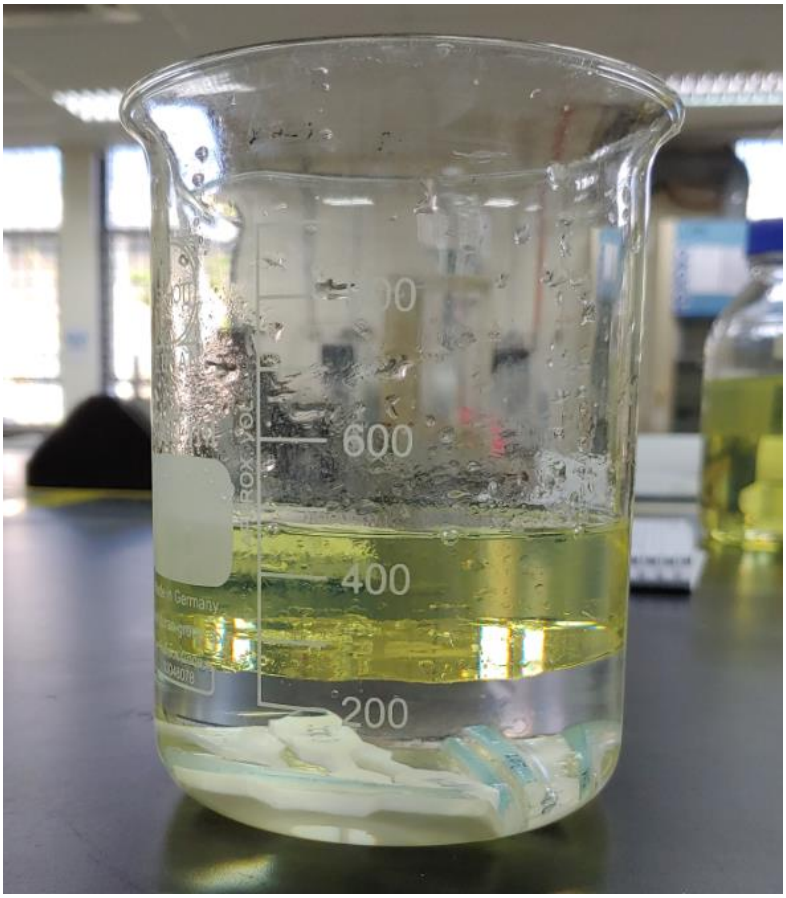

Figure 3.9 Mix solution of hydrocarbon and deionized water with 50% volumetric fraction.

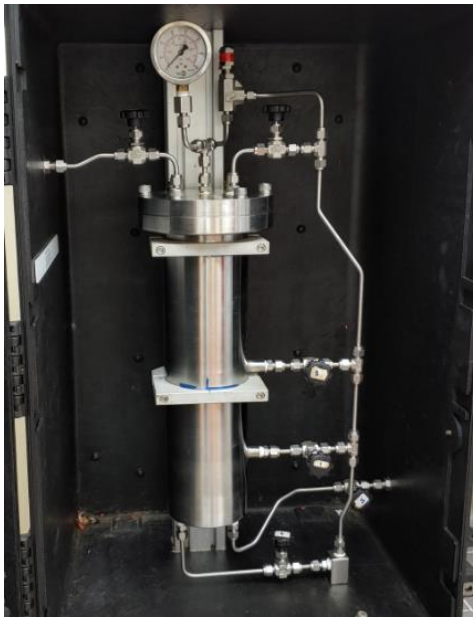

Figure 3.10 Mobile In-Situ Corrosion Monitoring (MiCorr) equipment for gas exposure test.

Figure 3.11 Autoclave equipment for gas exposure test.

3.5 Sample Naming

To prevent any mix up of specimens which may affect the test result, each specimens are labelled with unique name according to sample naming method shown in Table 3.4. The first character of the sample name specific the testing method and medium used to test the specimen. The second character of the sample name represent the required number of week for the test. The third character of the sample name specify the sample number.

TABLE 3.3 Sample Naming Method.

Medium Type	Week No.	Sample No.
A - H	1 - 3	1 - 5
E.g. A11 = Water Absorption Test, Distilled Water, Week 1, Sample No. 1		

3.6 Test Matrix

Table 3.5 shows the testing method used, medium, pH value, temperature and pressure condition, equipment used, test length and sample label of specimens in this study. All the experiments are planned and execute according to this test matrix.

TABLE 3.4 Test Matrix.

Testing Method	Medium	pH	Condition	Equipment	Test Length (Hour)	Sample Label
Water Absorption Test	Deionized Water	7.00	Room Temperature & Room Pressure	-	336	A21 – A25
	Sea Water	8.22	Room Temperature & Room Pressure	-	336	B21 – B25

	10% Acetic Acid	2.52	Room Temperature & Room Pressure	-	336	C21 – C25
	10% Nitric Acid	0.55	Room Temperature & Room Pressure	-	336	H21 – H25
Gas Exposure Test	CO_2 gas + Deionized Water + Hydrocarbon (50% Volumetric Fraction of Deionized Water)	-	25°C, 1 bar	MiCorr	168	D21 – D25
			25°C, 20 bars	MiCorr	168	F21 – F25
			45°C, 20 bars	Autoclave	120	G21 – G25
Control Unit	-	-	Room Temperature & Room Pressure	-	336	E21 – E25
Total Sample Required						40

Each pH value of medium for water absorption test are measured with a calibrated CyberScan pH 300 equipment. The equipment can be used to determine the pH value of solution in range of -2.00 to 16.00 pH and provide an accuracy within ± 0.01 pH. The pH value of each medium is shown in Figure 3.12 to 3.15.

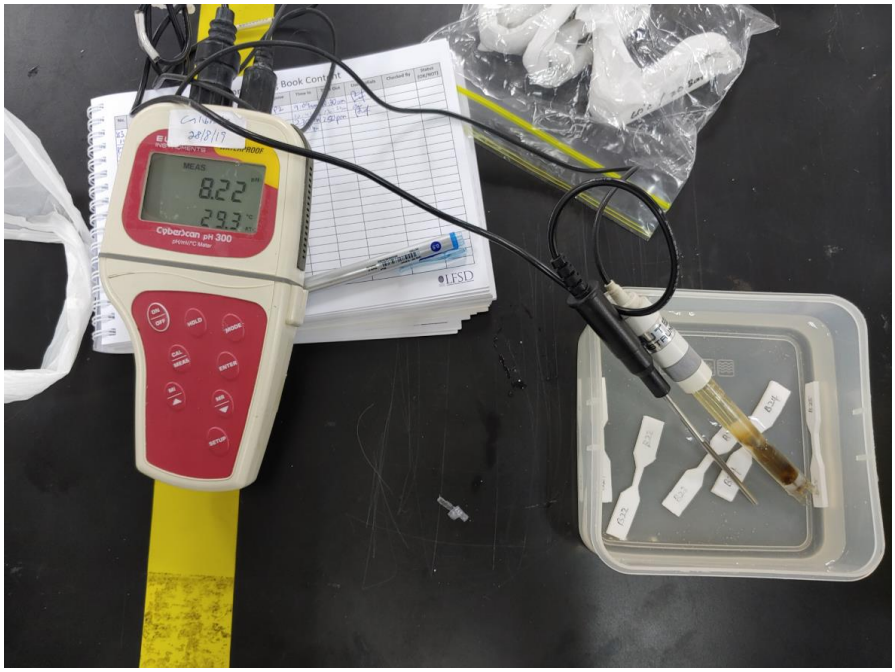

Figure 3.12 Sea water with pH 8.22.

Figure 3.13 10% acetic acid with pH 2.52.

Figure 3.14 10% nitric acid with pH 0.55.

Figure 3.15 Deionized water with pH 7.00.

3.7 Gantt Chart

Students are assigned with 28 weeks to complete their final year project. To be able to complete the project study within the time frame, all the activities need to be planned in correct time manner. Gantt chart tool is applied as to help student for better time management for the project as shown in Table 3.5 and Table 3.6.

FYP I Project Gantt Chart

TABLE 3.5 FYP I Project Gantt Chart.

Project Activities	**FYP I: Project Week**													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Selection of Project Titles														
Literature Review														
Progress Assessment I														
Proposal Defense														
Submission of Interim Draft Report														
Progress Assessment II														
Purchase of Resources														
Submission of Interim Report														

FYP II Project Gantt Chart

TABLE 3.6 FYP II Project Gantt Chart.

Project Activities	**FYP II: Project Week**														
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Specimen Preparation															
Water Absorption Test															
Gas Exposure Test															
Progress Assessment															
Report Drafting															
Final Report Submission															

Chapter 4
Results and Discussion

4.1 Results

Weight of specimens is measured and recorded from time to time after reached a specific time of immersion and gas exposure. A&D HR-250AZ weighing scale is used to measure the weight changes of specimen, equipment as shown in Figure 4.1. The equipment has a readability of 0.1 mg and linearity of ± 0.3 mg, which allow precision measurement for any changes in weight of specimen.

Figure 4.1 A&D HR-250AZ weighing scale.

Percentage Changes of Weight, W% is calculated using Equation 14.

$$W\% = \frac{W1-W0}{W0}\ x\ 100\% \qquad (14)$$

W% is the percentage changes of weight, W_1 (g) is the weight of the specimen at a given time, W_0 (g) is the initial weight of the specimen.

Before the water absorption test, initial weight of the specimens is weighted and recorded. After a specific period of immersion which are 24-hours, 168-hours and 336-hours, the specimens are removed from the medium and to be weighted again. Three reading are taken for each weighing process as to obtain an average value. This can help in obtaining a reading with higher accuracy and higher reliability. The readings are as shown in Table 4.1. Besides, Table 4.1 also shows the weight changes of specimen as compare to the initial weight reading, in terms of gram and %.

TABLE 4.1 Weight of Specimens Before and After Water Absorption Test (Initial Weight, 24-hrs, 168-hrs, 336-hrs).

Sample Label	Mass (g)					
	Before Test Start	Difference in Weight		24-hrs	Difference in Weight	
	Average	g chg	% chg	Average	g chg	% chg
A21	3.1069			3.1078	0.001	0.03%
A22	3.4827			3.4826	-0.0001	0.00%
A23	3.4729			3.4742	0.0013	0.04%
A24	3.4229			3.4236	0.0007	0.02%
A25	3.6017			3.6024	0.0006	0.02%
B21	3.2719			3.2729	0.0009	0.03%
B22	3.3907			3.3912	0.0005	0.01%
B23	3.569			3.5699	0.0009	0.03%
B24	3.8595			3.8609	0.0014	0.04%
B25	3.4199			3.4204	0.0005	0.01%
C21	3.505	-		3.506	0.001	0.03%
C22	3.5411			3.5427	0.0015	0.04%
C23	3.7995			3.8002	0.0007	0.02%
C24	3.619			3.6193	0.0003	0.01%
C25	3.2207			3.2213	0.0006	0.02%
E21	3.5026			3.5025	-0.0001	0.00%
E22	3.6064			3.6061	-0.0003	-0.01%
E23	3.1536			3.1535	-0.0002	-0.01%
E24	3.2867			3.287	0.0003	0.01%
E25	3.8826			3.8818	-0.0008	-0.02%

H21	3.755			3.7555	0.0005	0.01%
H22	3.3986			3.3994	0.0008	0.02%
H23	3.5453			3.5457	0.0004	0.01%
H24	3.3478			3.3487	0.0009	0.03%
H25	3.7091			3.7093	0.0002	0.01%

Sample Label	Mass (g)					
	168-hrs	Difference in Weight		336-hrs	Difference in Weight	
	Average	g chg	% chg	Average	g chg	% chg
A21	3.1074	0.0005	0.02%	3.1073	0.0004	0.01%
A22	3.4822	-0.0005	-0.01%	3.482	-0.0007	-0.02%
A23	3.4733	0.0004	0.01%	3.4731	0.0002	0.00%
A24	3.4233	0.0004	0.01%	3.4232	0.0003	0.01%
A25	3.6017	0	0.00%	3.6018	0.0001	0.00%
B21	3.2724	0.0005	0.02%	3.273	0.001	0.03%
B22	3.3909	0.0003	0.01%	3.391	0.0003	0.01%
B23	3.5696	0.0005	0.01%	3.5696	0.0005	0.01%
B24	3.8591	-0.0004	-0.01%	3.8598	0.0003	0.01%
B25	3.4202	0.0003	0.01%	3.4207	0.0008	0.02%
C21	3.506	0.001	0.03%	3.5054	0.0004	0.01%
C22	3.5412	0.0001	0.00%	3.5414	0.0002	0.01%
C23	3.8007	0.0013	0.03%	3.8001	0.0006	0.02%
C24	3.6197	0.0007	0.02%	3.619	0	0.00%
C25	3.2212	0.0005	0.01%	3.221	0.0002	0.01%
E21	3.5024	-0.0002	-0.01%	3.5025	-0.0001	0.00%
E22	3.6063	-0.0001	0.00%	3.6061	-0.0003	-0.01%
E23	3.1534	-0.0003	-0.01%	3.1535	-0.0001	0.00%
E24	3.2875	0.0008	0.02%	3.2868	0.0001	0.00%
E25	3.882	-0.0006	-0.01%	3.8819	-0.0006	-0.02%
H21	3.7551	-0.0004	-0.01%	3.7551	0.0001	0.00%
H22	3.3992	-0.0002	-0.01%	3.3992	0.0006	0.02%
H23	3.5454	-0.0003	-0.01%	3.5455	0.0002	0.01%
H24	3.3481	-0.0006	-0.02%	3.3481	0.0003	0.01%
H25	3.7093	0.0001	0.00%	3.7092	0.0002	0.00%

Before the gas exposure test of D21 - D25 and F21 – F25 polytetrafluoroethylene specimens using MiCorr, the initial weight of the specimens is weighted and recorded. Next, the specimens are undergoing gas exposure test for 168-hours under with condition of room temperature with 1 bar pressure and 20 bar pressure. The weight of the specimens is recorded immediately after the test. After that, the tested specimens are left under room temperature and room pressure condition for 168-hours to observe any changes to their weight. All the weighing of the specimens is taken three times to obtain an average value. The readings are as shown in Table 4.2.

TABLE 4.2 Specimen Weight Before and After Gas Exposure Test Under Room Temperature and Pressure of 1 Bar and 20 Bar using MiCorr for 168 Hours.

Gas Exposure Test Using Mobile In-Situ Corrosion Monitoring Equipment (MiCorr) (Room Temperature, Pressure of 1 Bar and 20 Bar)									
Sample Label	Mass (g)								
	Before Test Start	Difference in Weight		168-hrs	Difference in Weight		168-hrs After Test (Room Condition)	Difference in Weight	
	Average			Average			Average		
		g chg	% chg		g chg	% chg		g	%
D21	3.356	-		3.3591	0.0031	0.09%	3.3570	0.0010	0.05%
D22	3.455			3.4582	0.0032	0.09%	3.4561	0.0011	0.05%
D23	3.4922			3.4957	0.0035	0.10%	3.4935	0.0013	0.06%
D24	3.5102			3.5130	0.0028	0.08%	3.5110	0.0008	0.04%
D25	3.465			3.4679	0.0029	0.08%	3.4661	0.0011	0.05%
F21	3.4538			3.4775	0.0237	0.69%	3.4569	0.0031	0.15%
F22	3.5834			3.6079	0.0245	0.68%	3.5866	0.0032	0.16%
F23	3.3095			3.3321	0.0226	0.68%	3.3131	0.0036	0.18%
F24	3.5235			3.5475	0.0240	0.68%	3.5267	0.0032	0.16%
F25	3.1619			3.1845	0.0226	0.72%	3.1658	0.0039	0.20%

G21 – G25 polytetrafluoroethylene specimens are tested using Autoclave for 120-hours, with condition of 45 ^{o}C and 20 Bar pressure. The initial weight of the specimens is recorded before the test start and weighted again immediately after the test end. Next, the specimens are left in room temperature and room pressure condition for another 168-hours and the weight of the specimens is recorded again. All the readings are taken three times and to obtain an average value for better accuracy and higher reliability. The readings are recorded in Table 4.3.

TABLE 4.3 Specimen Weight Before and After Gas Exposure Test Under Temperature and Pressure of 45^{o}C and 20 Bar using Autoclave for 120 Hours.

Gas Exposure Test Using Autoclave (Average Temperature of 45^{o}C, Pressure of 20 Bar)									
Sample Label	Mass (g)								
	Before Test Start	Difference in Weight		120-hrs	Difference in Weight		168-hrs After Test (Room Condition)	Difference in Weight	
	Average			Average			Average		
		g chg	% chg		g chg	% chg		g	%
G21	3.5342	-		3.5514	0.0172	0.49%	3.5389	0.0047	0.23%
G22	3.6443			3.6632	0.0189	0.52%	3.6497	0.0055	0.27%
G23	3.6247			3.6439	0.0192	0.53%	3.6297	0.0050	0.25%
G24	3.467			3.4852	0.0182	0.53%	3.4722	0.0052	0.26%
G25	3.6389			3.6580	0.0191	0.53%	3.6443	0.0054	0.27%

Figure 4.2 shows the plotting of % changes of specimen weight versus hour of water absorption test. Different shape and colors of points represents the label of the specimen which immersed in different medium. The W% plot for the points are started with 0% as no weight changes at time zero. Weight changes of specimens is plotted at period of 24-hours, 168-hours and 336-hours.

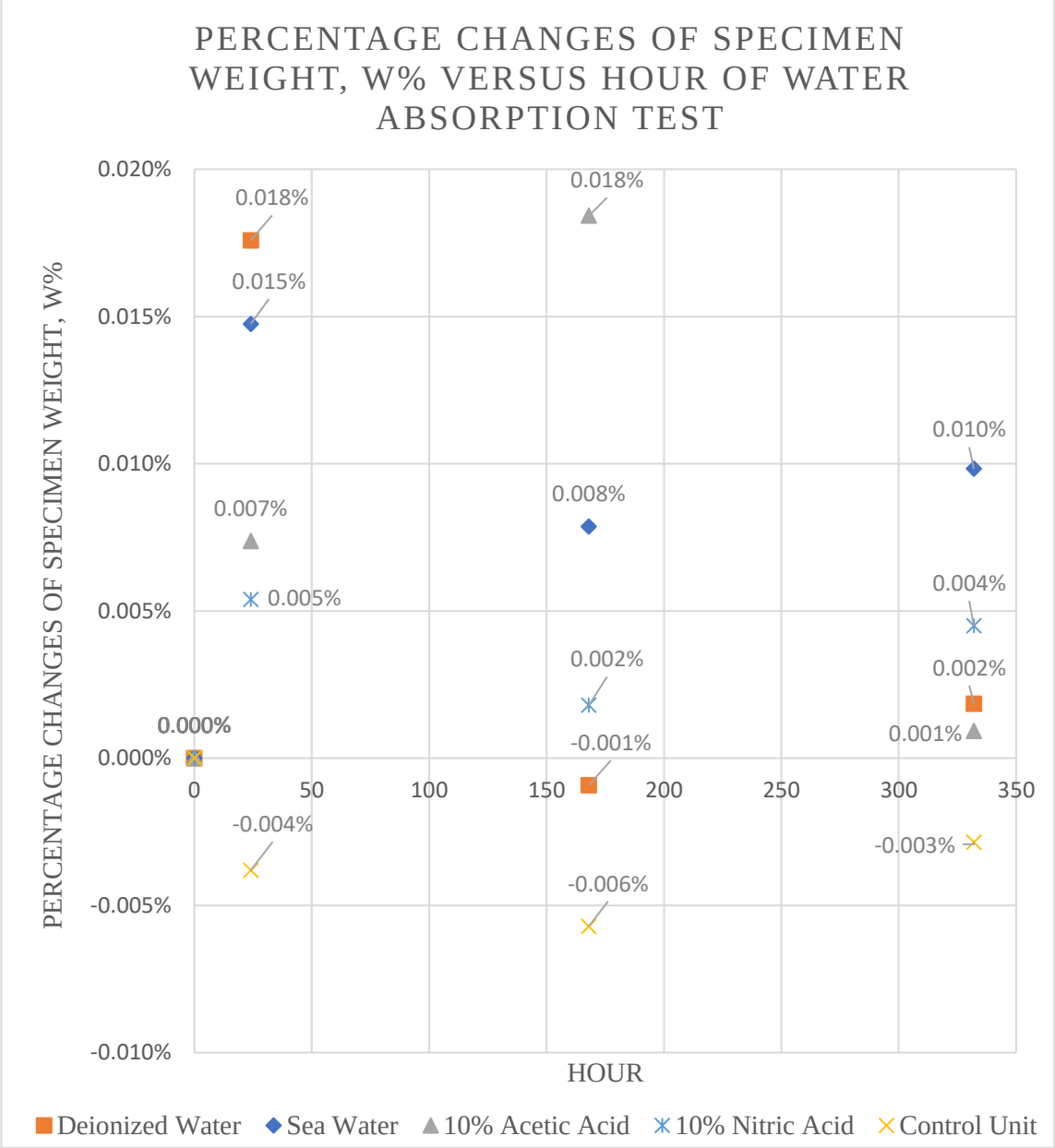

Figure 4.2 Chart of percentage changes of specimen weight, W% versus hour of water absorption test.

Figure 4.3 shows the plotting of changes of specimen weight versus hour of gas exposure test. Each color of line represents different gas exposure test condition as listed next to the graph. The W% plot is started with 0% as no weight changes at time zero. Weight changes of specimens is plotted at period of 168-hours and 336-hours for test

using MiCorr equipment and plotted at 120-hours and 288-hours for test using Autoclave equipment.

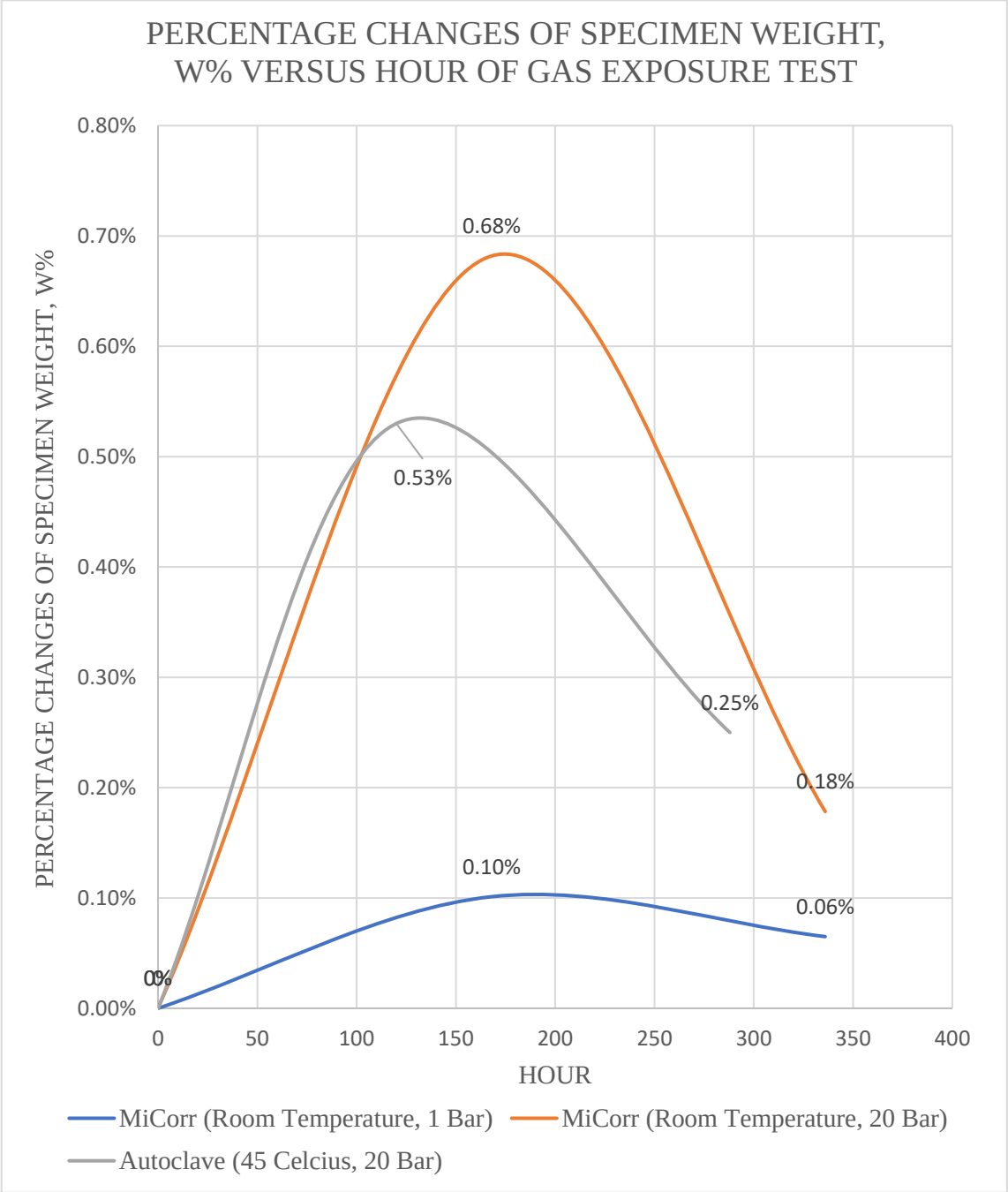

Figure 4.3 Chart of percentage changes of specimen weight, W% versus hour of gas exposure test.

4.2 Discussion

For water absorption test of PTFE specimens immersed into distilled water, sea water, 10% acetic acid and 10% nitric acid, the weight percentage changes of the PTFE specimens are found to below 0.018%. The results obtained are very close to the water absorption value result of < 0.01% which is tested by different material manufacturer following standard ASTM D570, as listed in Table 2.2.

Next, PTFE specimens are found to have no significant weight loss after immersed in light alkaline which is sea water, and strong acid which are 10% acetic acid and 10% nitric acid for 336-hours of time.

For gas exposure test of PTFE under 3 different conditions, which are: 1) Room Temperature, 1 Bar Pressure, 2) Room Temperature, 20 Bar Pressure and 3) 45^{o}C Temperature, 20 Bar Pressure, significant changes to the weight of specimen is observed compare to water absorption test. As shown in Figure 4.3, PTFE specimen exposed under 45 ^{o}C temperature and 20 Bar pressure have a steeper slope compare to PTFE specimen exposed under room temperature and 20 Bar pressure for the first 100 hours. However, as PTFE specimen are only exposed for 120-hours in autoclave while exposed for 168-hours in MiCorr, therefore the weight changes of gray line are stopped at 0.53% of increment but the weight changes of orange line is further increase to 0.68%. Next, PTFE specimen exposed under same temperature, but different pressure also shows a different in the weight changes. PTFE specimen exposed to 20 bar pressure will have higher weight increase compare to PTFE specimen exposed to 1 bar pressure with both under the same room temperature condition. In general, weight of PTFE specimen tends to increase greater when in high temperature and high-pressure condition. High temperature and high-pressure condition also cause the specimen to be deform and bend, as shown in Figure 4.4.

Figure 4.4 Comparison of appearance between control unit (left-hand side) and specimen after 45^{o}C temperature and 20 Bar pressure gas exposure test (right-hand side).

The bending angle of specimens is measure using a digital protractor from the midpoint of the specimen to the edge of the specimen, as shown by red lines in Figure 4.5. The bend angle reading is recorded in Table 4.4.

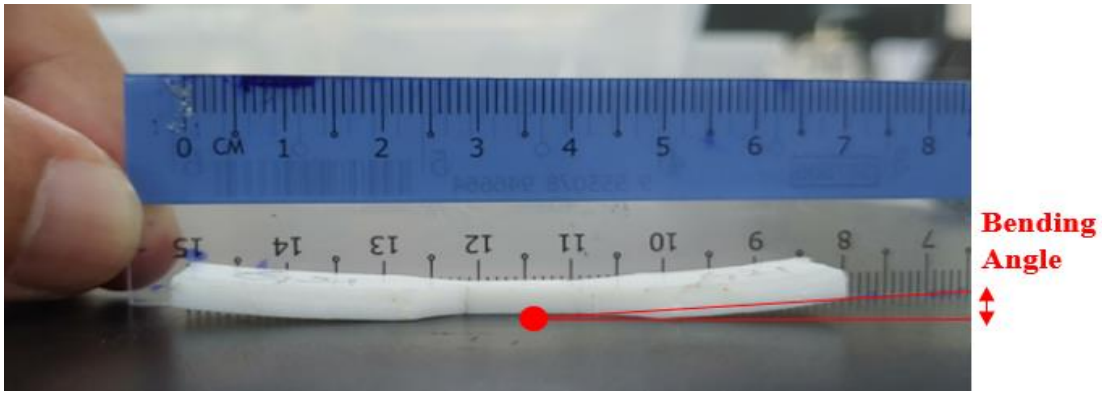

Figure 4.5 Illustrate on the bending angle of specimen after undergo gas exposure test.

TABLE 4.4. Bending Angle of Specimens.

Specimen Test Condition	Time when reading is taken	Bending Angle (Degree)
MiCorr (Room Temperature, 1 Bar Pressure)	168-hour after specimen is left under room temperature and room pressure condition.	1.2
MiCorr (Room Temperature, 20 Bar Pressure)		1.2
Autoclave (45 ^{o}C Temperature, 20 Bar Pressure)		1.2

After the gas exposure test, the specimens are left for 168-hours under room temperature and room pressure condition. The data listed in Table 4.3 shows specimens are observed to have drop in weight changes after left for 168-hours under room temperature and room pressure condition as compared to the weight measured right after the specimen has finished the gas exposure test.

PTFE can be concluded as chemically stable as no corrosion occur to the material even immersed in light alkaline and strong acid for 336-hours of period. No damage or flaws are observed from the surface of PTFE specimen after long period of immersion in the solution.

The weight changes of PTFE specimen due to gas exposure test are possibly caused by trapping of small particles such as CO_2 gas particles inside the PTFE molecular structure. High pressure may cause the small particles forced to be trapped inside the gap of PTFE structure and high temperature can increase the collision chance of small particles and lead to more small particles accidentally fit into the gap between the structure. Thus, there is a significant increase in weight of specimen after the specimens have exposed to high temperature and high-pressure condition compare the one exposed to lower temperature and lower pressure condition. Besides, after the specimen have been removed

from high pressure and high temperature condition, the small particles which are forced to fit into the gap of PTFE structure due to the pressure will be released back to the surrounding. Thus, the weight of specimens which has been tested with gas exposure test are noticed to be reduced after left under room temperature and room pressure condition for 168-hours.

Chapter 5
Conclusion and Recommendation

Polytetrafluoroethylene is classified as thermoplastic, which means the material has reversible chemical bond. Polytetrafluoroethylene can be remolded and recycled without affecting its original physical properties, make it manufacturable into industrial need.

Polytetrafluoroethylene exhibits very good resistance to corrosion and is described as chemically inertness, such properties is due to its strong attached bond between carbon and fluorine of its molecular structural. During the study, water absorption test is carried out to study the corrosive resistance of polytetrafluoroethylene using strong acidic solution to weak alkaline solution. Polytetrafluoroethylene specimens are immersed into different medium which are deionized water (7.00 pH), nitric acid (0.55 pH), acetic acid (2.52 pH) and sea water (8.22 pH). The weight of the specimens is measured and recorded for every interval period. The weight of the specimens is noticed to have minor changes within range of -0.006% to 0.018% which is very insignificant. The results observed is tally with the findings from literature review.

Besides, gas exposure test is carried out to understand on the possible reason of fracture of polytetrafluoroethylene. From the study, it is observed that the weight of polytetrafluoroethylene can be vary when the temperature and pressure is varied during gas exposure test. For the same length of exposure time, weight of polytetrafluoroethylene increases more when temperature and pressure are higher. Polytetrafluoroethylene are also observed to expand and bend after the gas exposure test.

For recommendation, from the literature review, after comparison of tensile strength result obtain from five different raw material manufacturer, tensile strength of

polytetrafluoroethylene is assumed in range of 20.59 to 36.77 MPa. From point of view of tensile strength, pure polytetrafluoroethylene might be damaged or scratch by impurities or solid particles when hydrocarbon is transporting at high velocity. Further study is required for method to enhance strength of polytetrafluoroethylene so the material can be applied to oil and gas field.

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